

## Standard Operating Procedure for:

GC4020 GE Co-Axial Detector and DSA 1000 Digital Spectrum  
Analyzer with 747 Series Lead Shield  
(Gamma Spec.doc)

Missouri State University

and

Ozarks Environmental and Water  
Resources Institute (OEWRI)

Prepared by: \_\_\_\_\_ Date: \_\_\_\_\_  
OEWRI Quality Assurance Manager

Approved by: \_\_\_\_\_ Date: \_\_\_\_\_  
OEWRI Director



**Table of Contents**

1	Identification of the test method .....	3
2	Applicable matrix or matrices.....	3
3	Detection Limit .....	3
4	Scope of the test method.....	3
5	Summary of test method.....	3
6	Definitions.....	3
7	Interferences.....	7
8	Health and safety.....	7
9	Emergency Procedures and Call List.....	8
10	Personnel qualifications .....	8
11	Equipment and supplies .....	9
12	Reagents and standards .....	9
13	Sample collection, preservation, shipment and storage .....	9
14	Quality control .....	9
15	Calibration and standardization .....	10
16	Procedure .....	10
17	Data acquisition, calculations, and reporting.....	14
18	Computer hardware and software .....	16
19	Method performance .....	16
20	Pollution prevention.....	17
21	Data assessment and acceptable criteria for quality control measures.....	17
22	Corrective actions for out-of-control or unacceptable data .....	17
23	Waste management.....	17
24	References .....	17
25	Bench sheet.....	19

## 1 Identification of the test method

Operation of the GC4020 GE Co-Axial Detector and DSA 1000 Digital Spectrum Analyzer with 747 Series Lead Shield to identify and quantify gamma-ray emitting radionuclides in soil samples. The gamma-ray spectroscopy system is a combination of the high-resolution germanium detector, 16K multi-channel analyzer, and the lead shield which is used to prevent high background counts.

## 2 Applicable matrix or matrices

This instrument can be used for natural soil samples.

## 3 Detection Limit

The detection limit for this system is 0.1 Bq or 5Bq. This method is not applicable to measure radionuclides that emit no gamma rays such as the pure beta-emitting radionuclides hydrogen-3, carbon-14, and strontium-90. This method is not applicable to measure Becquerel quantities of most elements having an atomic number greater than 92.

## 4 Scope of the test method

- 4.1 This procedure will be used as a laboratory reference guide for the collection of gamma-ray emitting radionuclide data from soil samples.
- 4.2 The instrument measures radionuclides in natural soil sediment samples that emit gamma-rays in quantities greater than 0.1Bq.
- 4.3 The procedures described here present principal components of the instrument, pre-operating instruction, actual sample analyses, instrument settings, safety components associated with ionizing radiation, and reporting procedures. The user should review the instrument manuals for further information about the instrument.

## 5 Summary of test method

- 5.1 The instrument manufacturer performed an energy calibration during installation. Standard Model 7503-7500 ML + Am-241 + Pb-210 was used for detector efficiency calibration. Regions of interest (ROI) were set to form OEWRI's specified library.
- 5.2 Sample mass is determined and the Marinelli beaker is placed over the detector's cryostat head. The multi-channel analyzer performs a Pulse Height Analysis that stores the isotope counts from the sample according to the energy that produced them. The Genie-2K V3.2 software locates and analyzes the peaks, subtracts background, identifies the nuclides, and corrects for parent/daughter interferences. A report is generated and activity is calculated in Bq/kg.

## 6 Definitions

- 6.1 ADC: Analogue to Digital Converter: This is an analog multiplexer that is the third electronic component within the gamma-spectroscopy system. It converts the analogue signal from the amplifier to a digital value.

- 6.2 Activity: Counts from a sample that accumulate at a given energy range due to genuine photon emissions from the sample. Higher sample activity decreases background input.
- 6.3 Amplifier: The second electronic component within the gamma-spectroscopy system that takes the pulse signal from the preamplifier and magnifies, filters, and shapes it to enhance the signal-to-noise ratio. The amplifier improves resolution and decreases response time to prevent overlap between pulses.
- 6.4 Analytical batch: The set of samples processed at the same time.
- 6.5 Background interference: Internal background interference would come from the detector that can contribute to gamma rays at the same energies as those in environmental samples. The detector is made from low-emitting gamma ray materials. External background interference would come from the laboratory environment and the shield associated with the system. The lead shield used for the OEWRI system is 12" thick and lined with copper.
- 6.6 Blank: The Marinelli beaker used to hold the sample during laboratory analysis. Only the Marinelli beaker is measured to determine additions or interferences present from apparatus or the laboratory environment.
- 6.7 Chain of Custody (COC): Used to describe the written record of the collection, possession and handling of samples. Chain of custody forms should be completed as described in the Chain of Custody SOP # 1030R01. Chain of custody (COC) forms are located on a board in Temple Hall 125.
- 6.8 Channel: An energy window; one of a multichannel analyzer's memory locations for storage of a specific level of energy.
- 6.9 Check Standard: Standard sediment sample of known concentration and purity that produce consistent concentrations of analytes. These standards are used to check instrument performance.
- 6.10 Detector: The first electronic component within the gamma-spectroscopy system that contains a charge-sensitive preamplifier that serves as an interface between the detector crystal and the pulse-processing and analysis electronics. The charge produced from the detector by the gamma radiation for the samples is integrated and amplified to produce a step-function pulse. The amplitude of that pulse is proportional to the total charge.
- 6.11 Efficiency: The number of impulses recorded by the detector with respect to the number of gamma photons actually emitted by the radiation source. The relative efficiency of the GC4020 model associated with this gamma-spectroscopy system is 40.

6.12 Efficiency Calibration: A function that correlates the net counts in the FEPs of each gamma ray obtained from a radionuclide standard to counts obtained from an unknown sample. Standard and sample counts occur after FEP position is established by energy calibration and ROIs are set. This calibration illustrates the detector's efficiency for capturing gamma rays of specified energies and allows quantification.

6.13 Energy Calibration: A function that correlates each channel in the displayed spectrum from the MCA with a specific unit of energy that identifies specific radionuclides. This calibration allows peaks to be identified by their location in the calibrated spectrum.

6.14 FEP: Full Energy Peak: The peak in an energy spectrum of gamma ray photons that occur when the full energy of the incident photon is absorbed by the detector.

6.15 Geometry: How the sample is presented to the detector. The OEWRI laboratory uses a Marinelli beaker (Lab Impex Systems: Model 530G-E) with a freeboard volume of 0.4L and endcap diameter of 7.6cm.

6.16 Isotope: One of two or more atoms of the same chemical element but with different atomic weights, so they have the same number of protons but different numbers of neutrons.

6.17 Laboratory Duplicate (LD): Two samples taken from the same sample bag at the same time and placed under identical circumstances throughout laboratory procedures. Analysis laboratory duplicates indicates the precision associated with laboratory procedures.

6.18 MCA: Multi-Channel Analyzer: The final electronic component within the gamma-spectroscopy system. It registers, or counts, the pulses that emerge from the ADC and sorts them in the channel that corresponds to the energy that produced the pulse.

6.19 Method detection limit (MDL): The lowest level at which an analyte can be detected with 99 percent confidence that the analyte concentration is greater than zero.

- a. To calculate the MDL:
- b. Prepare triplicates of two sediment samples with low organic matter. The laboratory director or supervisor will choose appropriate samples to use to determine MDL.
- c. Analyze all samples.
- d. Include all sample processing steps in the determination.
- e. Calculate the standard deviation ( $s$ ).
- f. From a table of the one-sided  $t$  distribution select the value of  $t$  for  $7 - 1 = 6$  degrees of freedom at the 99% level. This value is 3.14
- g. The product 3.14 times  $s$  is the desired MDL.

- 6.20 Minimum Quantification Interval: The lowest level that can be quantitated accurately and is generally defined as four times the method detection limit = 4(MDL).
- 6.21 Nuclide: A general term applicable to the isotopes of all elements, including both stable and radioactive forms.
- 6.22 Nuclide Identification (NID): The process of identifying radionuclides by comparing peak energies detected with entries in a nuclide library.
- 6.23 Peak: A statistical distribution of digitized energy data for a single energy.
- 6.24 Peak Channel: The channel number closest to the centroid of a peak.
- 6.25 Peak Fit: The optimization of parameters to match an expected model shape to empirical data (Gaussian fit) typically performed using a least squares method.
- 6.26 Pulse Height Analysis (PHA): The acquisition of energy-correlated data in the multichannel analyzer. Each channel (energy window) is incremented by one count for each event that falls within the window, producing a spectrum that correlates the number of energy events as a function of their amplitude.
- 6.27 Radionuclide: A radioactive isotopes.
- 6.28 Regions of Interest (ROI): A user-defined area of the spectrum which contains data of particular interest such as peaks associated with isotopes.
- 6.29 Relative Percent Difference (RPD): calculated as the difference between a sample and duplicate results, divided by the average of the sample and duplicate results, multiplied by 100%.
- 6.30 Sample mass: Increase in sample mass generally increases the total number of photons emitted by the sample and potentially captured by the detector although sample self-attenuation can occur with larger sample mass. Sample self-attenuation, where low-energy photons emitted from the sample are absorbed within the sample matrix itself and are not picked up by the detector, are particularly detrimental to  $^{210}\text{Pb}$  determinations.
- 6.31 Smoothing: When the content of each channel is replaced by a weighted average over a number of adjacent channels to decrease the effects of statistical uncertainties in computerized spectrum analysis.
- 6.32 Spectrum: A distribution of radiation intensity as a function of energy or time.
- 6.33 Uncertainty: The lack of complete knowledge of a sample's decay rate due to the random nature of the decay process and the finite length of time used to count the sample.

6.34 Window: A term describing the upper and lower limits of radiation energy accepted for counting by a spectrometer.

**7 Interferences**

7.1 Precision and accuracy can be affected by the efficiency of the detector, the activity of the sample, the mass of sample, the geometry used in presenting the sample to the detector, internal and external background interferences, and the quality and thickness of lead shielding.

7.2 Background interference: Internal background interference would come from the detector that can contribute to gamma rays at the same energies as those in environmental samples. The detector is made from low-emitting gamma ray materials. External background interference would come from the laboratory environment and the shield associated with the system. The lead shield used for the OEWRI system is 12" thick and lined with copper.

**8 Health and safety**

8.1 Researchers, faculty members, staff members, and students will use germanium detection instrumentation in accordance with their departments' established procedures and the requirements of this standard practice. Only trained personnel are allowed to operate this instrument.

8.2 The responsible person for this instrument is the director of the Ozarks Environmental and Water Resources Institute.

8.3 Liquid nitrogen's temperature is -196°C and it can cause frostbite if not handled properly. Avoid skin contact with liquid nitrogen or with surfaces cooled by liquid nitrogen. Wear protective clothing including gloves, protective booties for exposed feet, and safety goggles. Items should fit loosely so that they can be thrown off quickly if liquid should spill or splash into them. Always handle liquid nitrogen in well-ventilated areas. Remove metal jewelry/watches on hand and wrists.

8.4 There are pressurized gas lines associated with this instrument, always use eye protection. Use caution when connecting and disconnecting the lines. Use two wrenches when connecting or disconnecting gas line couplings to avoid loosening the bulkhead coupling. Check the condition of the gasket seal on the plug portion of each coupling. Remove any metal slivers or debris that could compromise the seal.

8.5 The Model 7503-7500ML + Am-241 + Pb-210 was prepared by Eckert & Ziegler Isotope Products. The standard is in a plastic 500 ml Marinelli beaker (530G-E) and is distributed in a 1.7 g/cc sand matrix. A standard wipe test was performed to ensure that the source was not leaking prior to shipment. The source was wiped over its entire surface with a moistened filter paper disk. The filter was dried and checked for activity using a scintillation detector. There was <0.001  $\mu$ Ci beta-gamma and <0.0001  $\mu$ Ci alpha of removable activity. A hand-held scintillation detector was used by the Missouri State University Radiation Safety Officer to check the standard for leaks upon arrival and no leaks were found. Radiation

safety surveys are standard protocol for the University and each survey performed in Temple Hall 125 produced compliance. The Missouri Radiation Control Program (MRCP) inspection on April 20, 2009 found that all regulated machines/materials were in general compliance with the Missouri Department of Health and Senior Services and MRCP protocol for minimum radiation safety. The standard is stored within a labeled drawer within Temple Hall. The efficiency calibration was performed immediately after receiving the standard, so additional calibrations are not necessary. Only the technician associated with the gamma spec system should use the standard, but due to decay processes the standard is really only used once during the efficiency calibration so additional handling is not necessary.

- 8.6 Instrument use and instrument service records will be maintained for two years or until the ownership of the instrument is transferred or the instrument is decommissioned. If the instrument is transferred to non-MSU persons, that transfer must be reported to regulatory agencies and the OIA representative. The QA/QC manager will maintain records and report necessary information regarding the instrument.
- 8.7 Never attempt to override any safety feature

## **9 Emergency Procedures and Call List**

- 9.1 Partial Liquid Nitrogen First Aid:
  - a. If a person seems to become dizzy or loses consciousness while working with liquid nitrogen, move to a well-ventilated area immediately. If breathing is difficult, give oxygen and call 911. If breathing has stopped, apply artificial respiration and call 911.
  - b. If exposed to liquid or cold gas, restore tissue to normal body temperature 98.6° F (37° C) as rapidly as possible, followed by protection of the injured tissue from further damage and infection. Remove or loosen clothing that may constrict blood circulation to the frozen area. Rapid warming of the affected part is best achieved by using water at 108° F/42° C). Under no circumstances should the water be over 112° F/44° C, nor should the frozen part be rubbed either before or after re-warming. Call 911 if necessary.
- 8.8 Turn lines off immediately when a liquid nitrogen leak from the source tank is observed. If the leak is coming from the handle attempt to re-tap the handle. Contact the laboratory manager 836-3198 or Prax Air 417-869-0544 if the leak continues or if the source tank appears to be damaged or not venting.
- 9.2 Questions and additional instruction should be directed to the laboratory manager at 417-836-3198.

## **10 Personnel qualifications**

Soil parameters will be collected by Missouri State University (MSU) graduate assistants who have received appropriate training, prior coursework, and field experience regarding the collection of soil parameter data, and who are familiar with all of MSU's sample handling and labeling procedures. All operators of the GC4020 GE Co-Axial

Detector and DSA 1000 Digital Spectrum Analyzer will be trained and will know all safety and operation procedures before using the instrument.

**11 Equipment and supplies**

- 11.1 GC4020 GE Co-Axial Detector, DSA 1000 Digital Spectrum Analyzer, Vertical Slim Line Dipstick Cryostate, and top-opening lead shield.
- 11.2 Liquid Nitrogen source tank – Prax Air NI LC 160-22
- 11.3 500 ml plastic Marinelli beaker (530G-E)

**12 Reagents and standards**

- 12.1 Model 7503-7500ML + Am-241 + Pb-210 Eckert & Ziegler Isotope Products Standard for Pb-210, Am-241, Cd-109, Co-57, Te-123m, Cr-51, Sn-113, Sr-85, Cs-137, Y-88, Co-60 (energy 1173 keV), Co-60 (energy 1333 keV), and Y-88.

**13 Sample collection, preservation, shipment and storage**

Sediment samples arrive to the laboratory in plastic bags. There are no special provisions for shipment. Portions are transferred to a Marinelli beaker prior to analysis. There are no special provisions for storage.

**14 Quality control**

- 14.1 Quality control program: The minimum requirements of the quality control program for this analysis consist of an initial demonstration of laboratory capability and the periodic analysis of blanks and standard check samples as a continuing check on performance. The laboratory must maintain performance records that define the quality of the data that are generated.
  - a. Analyses of laboratory blanks are required to demonstrate freedom from contamination.
  - b. The laboratory shall, on an ongoing basis, demonstrate through calibration verification and analysis of the ongoing precision and recovery sample that the analysis system is in control.
  - c. The laboratory should maintain records to define the quality of data that is generated.
- 14.2 Initial demonstration of performance. The following must be satisfied before the analytical procedure may be used for samples and before a new analyst may analyze samples.
  - a. Method Detection Limit (MDL) – To establish the ability to detect the analyte, the analyst shall determine the MDL by carrying through 7 or more blanks (Marinelli beaker) through the analytical procedures. The average value,  $X$ , and the standard deviation of the values,  $s$ , shall be calculated. The MDL is equal to  $3s$  ( $3 \times$  standard deviation). A 20 hour count of the vacant shield produced no activity for the nuclides of interest, so the MDL is 0.0 Bq/unit/20hours for the nuclides listed for Standard Model 7503-7500ML + Am-241 + Pb-210 from Eckert & Ziegler Isotope

Products.

b. Initial Precision and Recovery – To establish the ability to generate acceptably precise and accurate results, the manufacturer performed an extensive calibration of the instrument using various energy sources. The energy calibration should remain stable for the life of the instrument. The efficiency calibration was performed when the standard was received and should remain stable for the life of the instrument for the listed isotopes.

## 15 Calibration and standardization

15.1 Energy calibration was completed by the manufacturer, Canberra. The calibration certificate is kept with original operating and safety manuals in the OEWRI office.

15.2 Efficiency calibration was completed by OEWRI staff and should remain stable for the life of the instrument. Isotopes, associated gamma-ray energy, half-life, and activity information are listed in the certificate of calibration multi-nuclide standard source sheet from the manufacturer.

15.4 The lead shield should minimize accuracy errors and interferences from the laboratory setting.

15.5 Any damage to the instrument will be reported to the QA/QC manager. The instrument will be maintained as defined in the user manual.

15.6 A 20 hour count of the vacant shield produced no activity for the nuclides of interest, so the MDL is 0.0 Bq/unit/20hours for the nuclides listed for Standard Model 7503-7500ML + Am-241 + Pb-210 from Eckert & Ziegler Isotope Products

## 16 Procedure

### 16.1 Pre-analysis Procedures

a. Software Startup

1. Turn on the computer and log on with the correct user name and password.
2. Double click on the Gamma Acquisition & Analysis desktop icon to start the program.
3. Two windows will open, one is the Genie 2000 VDM window and the other is the Gamma Acquisition & Analysis window. The Genie 2000 VDM window cannot be opened, but the Gamma Acquisition & Analysis window can.
4. Click **File** and then click **Open datasource** within the Gamma Acquisition & Analysis window. Ensure that the “Source:” is on “Detector” in the Open Datasource dialogue box illustrated in Figure 1 below. If the “Source:” is not on “Detector”, click the Detector radial button. Then click **DET01** and then **Open**.

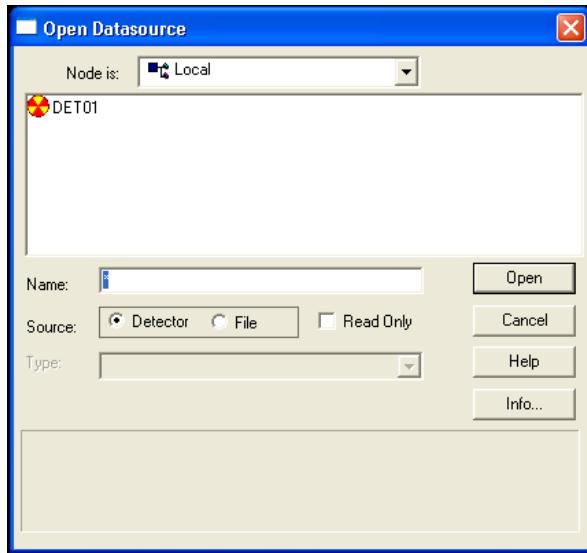


Figure 1. Open Datasource Dialog Box.

5. The title bar should change to "Gamma – DET01" and the Spectral Display should be black with no peaks as illustrated in Figure 2 below. If peaks are present, click the Clear button.

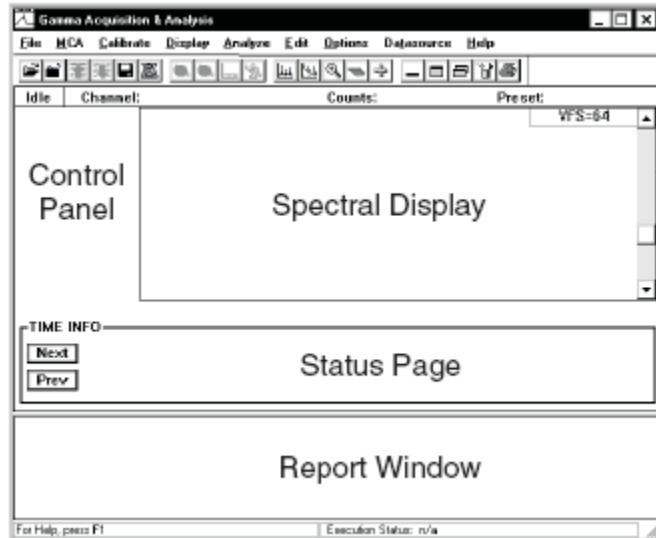


Figure 2. Parts of the Acquisition and Analysis

## 16.2 Instrument Checks

- a. Check the status of the Canberra DSA-1000 Multichannel Digital Spectrum Analyzer (on the benchtop, right of the computer). Figure 3 illustrates the front panel of the Spectrum Analyzer. All of the lights on the front panel should be green except two of the voltage meter bars.

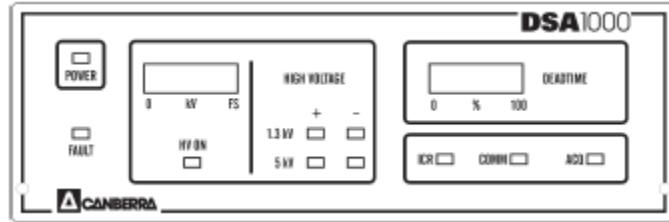


Figure 3. DSA – 1000 Front Panel

- b. Check the main unit's crystal detector by opening the top of lead shield by sliding it counterclockwise. Look for a green light at the bottom of the cryostat head. If only a yellow light is present or a flashing green light is present, contact the laboratory manager immediately and do not continue with the analyses.
- 16.3 Sample Preparation
  - a. Label a Marinelli beaker with a sample ID. Weigh that beaker and record the weight. Fill that beaker fully with the sediment sample, tap the beaker on the benchtop three times to compact the sample, re-weigh, and record that weight on the bench sheet. Specific compaction and sample volume will be dependent on the project and the volume of sample available.
  - b. Open the top of the lead shield by sliding the top counterclockwise.
  - c. Place the Marinelli beaker over the detector's cryostat head.
  - d. At the computer, click **Edit** and then **Sample Info**. Type the Sample Title, Collector Name, Sample Description, Sample ID, Type, and Sample Geometry in the Edit Sample Information dialogue boxes. Click **OK**.
- 16.4 Sample Analysis
  - a. Ensure that the "Preset live" time at the top of the window is "72000/0.00" and that the spectral display is clear. If the display is not clear, click the **Clear** button in the Control Panel area. Note: some of the live times may vary depending of the project; follow live times noted on the COC.
  - b. Make sure the Interactive NID is set correctly to the specified library by clicking **Options** and **Interactive NID**.... Check that the top of the window it reads: "Library: C:\GENIE2K\ CAMFILES\New MSU.NLB". If it does not, click **Setup** and **Select** in the Interactive NID Setup dialogue box. Navigate to the CAMFILES folder and select the mentioned library. Click **OK** in the Open dialogue box. Then click **OK** and **Exit** in the Interactive NID Setup dialogue box.
  - c. Click **Save**.

- d. Regions of interest (ROI) can be viewed by clicking the **Expand On** button in the Control Panel area and then selecting the minus (previous) or plus (next) buttons under the ROI Index.
  - 1. To load preset ROIs from a file, click **Display**, then **ROIs**, and then **Load**. Select the NewMSU.ROI file from the dialogue box and click **OK**.
  - 2. To create ROIs, move the left and right marker to a desired location by clicking on the spectral display, and then using “Ctrl+L” for the left marker and “Ctrl+R” for the right marker and then pushing the Insert key on the keyboard.
  - 3. To delete ROIs, move the markers as previously described around the ROI and push the Delete key on the keyboard.

#### 16.5 Post Sample Analysis Shut Down

- a. The analysis will stop automatically after the preset time has been reached. If need be, the analysis can be stopped at anytime during the analysis by clicking the **Stop** button under the word **Acquire** in the Control Panel region of the Gamma Acquisition & Analysis window (see Figure 4).
- b. Remove the Marinelli beaker from the detector.
- c. Click **File Save As...** and name the file accordingly.
- d. Click the Close Data Source button. Then click Open Data Source button and retrieve the newly saved file.

#### 16.6 Post Sample Analysis Data Processing and Report

- a. Ensure that the detector screen is not displayed. Create a report for the data collected by clicking **Analyze**, then **Execute Sequence**, and then **NID+ w/Report** or another report generating sequence specific to a project.
- b. Click on the **Maximize the Report Window** hot button  to see the analysis processed report or click on the **Restore Report Window to Default Size** hot button  to view both the analysis and the report. To return to just the Spectral Display of the analysis, click the **Minimize the Report Window**  hot button.

#### 16.7 Export and Save Data

- a. Save the analyzers data to the hard drive by clicking **Save**.
- b. A \*.rpt file is simultaneously created when the data is saved as a \*.CNF file. The \*.rpt file is located in the REPFILES folder. To access a \*.rpt file, go to the desktop, double click the **My Computer** icon, double click the **Local Disk (C:)** icon, double click the **GENIE2K** folder, and double click the **REPFILES** folder. The file should be there with the same name as the \*.CNF file.

- c. To export a report to a Microsoft Office Excel file cut, paste, and edit as directed during training.
- d. Save the excel file using the same name as in prior steps.

## 17 Data acquisition, calculations, and reporting

17.1 The Genie 2000 3.1 software program identifies nuclides using matrix formalism similar to the one developed in SAMPO80 that uses the least square fitting method with cubic B-spline basis functions which reduce the influence of statistical fluctuations in the gamma ray spectra.

- a. The least square method gives the best-fit curve that has the minimal sum of the deviations squared (*least square error*) for each channel. Once the best-fit curve is obtained, the smoothing end condition is determined automatically using Reinsch's criterion which eliminates the initial smoothing input value that is difficult to determine prior to actual calculations. The cubic B-spline method eliminates fluctuation noise sufficiently with little distortion and smoothes the resultant curve which increases peak distinction when peaks overlap. The result is smooth gamma ray spectrum data with distinctive peaks in each channel.
- b. The software program compares each nuclide in the analysis library against the observed peaks and builds a matrix of possible identifications using specific criteria that takes into account all lines of a nuclide with their proper branching ratios. Nuclides that pass through these three tests with a confidence index greater than 0.30 are classified as identified.
  1. A penalty function is applied to the nuclide confidence value that considers the difference between the reference peak energy and the measured peak energy. Increased difference between the reference and measured energies reduces the confidence value.
  2. The confidence value is reduced further by additional functions that consider the branching ratio of the peak, efficiency of the energy corrected for attenuation, and mass attenuation.
  3. Then the confidence values is multiplied by the decay time penalty function that removes nuclides from the calculation that have too short a half-life to be probable and considers elapsed time and nuclide half-life.

17.2 The activity per unit mass of the sample, C, is calculated as

$$C = \frac{S}{V \epsilon' y T_1 U_f K_c K_w}$$

where

S = the net peak area

$V$  = the sample mass (or volume)  
 $\epsilon'$  = the attenuation corrected efficiency (page 334 of the manual)  
 $y$  = the branching ratio of the peak energy  
 $T_1$  = the live time of the count (analysis) in seconds  
 $U_f$  = the conversion factor for the correct units of activity  
 $K_c$  = the correction factor for the nuclide decay during counting  
(page 335 of the manual)  
 $K_w$  = the correction factor for the nuclide decay from the time the  
sample was obtained to the start of the count (page 335 of  
the manual)

17.3 The random uncertainty of the activity,  $\sigma_C$ , is a combined weighed standard deviation of random aspects of gamma ray spectra analyses. Those aspects include random uncertainty as well as the uncertainty of the net peak area, sample quantity, effective efficiency, branching ration, composite decay correction factor, and all of the correction factors as described on pages 337-338 in the manual.

17.4 The total uncertainty of the activity,  $C$ , is calculated as

$$\sigma_{C(T)} = \sigma_C + \frac{\sigma_{sys} \cdot C}{100}$$

where

$\sigma_{sys}$  = the user defined systematic uncertainty (%)

17.5 After a nuclide is identified an interference calculation occurs where identified nuclides are searched for possible interference sets. An interference set is defined as two or more nuclides with at least one common peak that the peak search and area calculation phases have not been able to resolve into multiple peaks. The activities of these nuclides are calculated as a solution to a linear least squares equation. The activity of a nuclide that is not part of an interference set is calculated as a weighed average of the activities calculated for each one of its peaks unless the lines have been marked as excluded form this weighted average.

17.6 The Parent-Daughter Correction algorithm first examines each nuclide as a potential daughter. Each potential daughter is examined to see if a specified parent exists and if the parent was identified in the sample spectrum. The corrected daughter activity and its uncertainty is calculated from the uncorrected daughter activity, the weighted mean parent activity, and the respective uncertainties of both. The equation is defined on pages 343-345 of the manual.

17.7 Minimal Detectable Activity (MDA) is calculated for both the identified and unidentified radionuclides using methods developed by L.A. Currie.

17.8 Reporting results: Results should be reported to 0.1 Bq/kg precision.

17.9 Relative percent difference (RPD):

$$\left[ \frac{(A-B)}{\text{AVERAGE}(A,B)} \right] 100$$

A = original sample concentration  
B = duplicate sample concentration

17.10 Standard Deviation: The evaluation of MDL and precision require calculation of standard deviation. Standard deviations should be calculated as in equation 2.

$$s = \left\{ \frac{\sum x^2 - [(\sum x)^2/n]}{n-1} \right\}^{1/2}$$

Where: n = number of samples,  
x = concentration in each sample.

Note: This is the sample standard deviation calculated by the STDEV function in Microsoft Excel.

17.11 Coefficient of Variation (Cv%): The evaluation of accuracy require the calculation of coefficient of variation which is the standard deviation of the multiple sample measurements divided by the mean of those measurements.

$$Cv\% = (s/\bar{x})100$$

## 18 Computer hardware and software

18.1 Genie 2000 3.1 and 3.1A by Canberra Instruments were purchased for the instrument for general operating and communication.

18.2 Genie 2K Gamma Spec V3.2 by Canberra Instruments was purchased for actual analysis and formatting of the data produced.

18.4 This document is created using Microsoft Word. The Word file name for this SOP is: Gamma Spec.doc

18.5 Microsoft Excel is used for recording and reviewing the final formatted data from the instrument.

## 19 Method performance

19.1 The desired performance criteria for this measurement are:

- Detection limit:  $\leq 0.01$  Bq/kg for each isotope
- Precision:  $\pm 20\%$
- Accuracy:  $\pm 10\%$
- Minimum Quantification Interval: 0.01 Bq/kg

19.2 A 20 hour count of the vacant shield produced no activity for the nuclides of interest, so the MDL is 0.0 Bq/unit/20hours for the nuclides listed for Standard Model 7503-7500ML + Am-241 + Pb-210 from Eckert & Ziegler Isotope Products.

**20 Pollution prevention**

No wastes requiring disposal are generated from these procedures.

**21 Data assessment and acceptable criteria for quality control measures**

21.1 The analyst should review all data for correctness.

21.2 Relative percent difference (RPD) should be calculated for pairs of duplicate analyses to determine precision. The desired precision is  $\pm 20\%$

21.3 The desired accuracy is  $\pm 10\%$

21.4 The completed Excel spreadsheet is reviewed by the analyst's supervisor or the OEWRI QA officer.

**22 Corrective actions for out-of-control or unacceptable data**

22.1 The results for precision and blank data are compared to the acceptable values for this analysis;  $\pm 20\%$  and 0.0 Bq/kg for all isotopes, respectively.

22.2 If data are unacceptable for any reason, the analyst should review their analytical technique prior to conducting this analysis again.

22.3 Precision and accuracy can be affected by the efficiency of the detector, the activity of the sample, the mass of sample, the geometry used in presenting the sample to the detector, internal and external background interferences, and the quality and thickness of lead shielding. OEWRI always uses Marinelli beakers for sample analysis to optimize detection and recommends that enough sample volume is collected in the field to fill the Marinelli beaker to address potential non-detections in data sets. The lead shield associated with the OEWRI system is 12" thick and lined with copper to minimize external background interferences and the interior of the shield is wiped out as necessary to minimize internal interferences.

22.4 The instrument may require trouble shooting techniques if the data are unacceptable. All instrument maintenance will be conducted by the QA/QC manager or sent to the manufacturer.

**23 Waste management**

No wastes generated in this method are hazardous. Sediment samples are returned to the client. Beakers are washed and reused for analyses or rinsed and recycled properly.

**24 References**

24.1 19 CSR 20, "Radiation Protection", Missouri Department of Health and Senior Services.

- 24.2 29 CFR Part 1910, Occupational Safety and Health Administration.
- 24.3 Missouri Statutes Section 192.400, "Radiation Control", Department of Health and Senior Services.
- 24.4 User's Manual. 2003. Canberra Industries, Inc., Meriden, CT.
- 24.5 Genie 2000 3.1 Customization Tools Manual. 2006. Canberra Industries, Inc., Meriden, CT.
- 24.6 SPI Liquid Nitrogen Dewars & Supplies: Instructions for safe handling in the microscopy laboratory, <http://www.2spi.com/catalog/instruments/nitrodew-supp.html>

**25 Appendices**

See page 19 for the bench sheet. The analyst should make a copy of this form for each analytical batch and should report these weights to the QA/QC manager when transferring data.

Missouri State University  
Ozarks Environmental and Water Resources Institute  
Springfield, Missouri  
Gamma Spec Mass Data Sheet

Analyst: \_\_\_\_\_

Data reviewed by:

Date: \_\_\_\_\_

Project: \_\_\_\_\_

**Comments:**